CHAPTER III
EXPERIMENTAL

3.1 Materials and Instruments

3.1.1 Chemicals

Gelatin from porcine skin, low gel strength (Fluka)

Gelatin from porcine skin, medium gel strength (Fluka)

Gelatin friom porcine skin, high gel strength (Fluka)

Glutaraldehyde (Sigma Aldrich, ACS reagent 50 wt.%)

3.1.2 Equipment

Thermalgravimetry analysis or TGA (Perkin Elmer, TGA7)

Melt rheometer (Rheometric Scientific, ARES)

beaker

Flask

Magnetic bar

Ultrasonic bath

Petri dish

3.2 Experimental

3.2.1 Preparation of Gelatim Films

Glutaraldehyde–gelatin crosslinked films (GTA–Ge) were prepared by adding the appropriate volume of GTA solution to the gelatin solution (10 vol.%), with GTA concentrations varying from 0.5 to 7 vol%. Non-crosslinked gelatin films (Ge) were prepared from an aqueous gelatin solution (10%, v/v) at 50 °C and under continuous stirring for 40 min. The GTA-Ge and Ge solutions were poured into plastic petri dishes (10 cm of diameter) . Crosslinked films were obtained after allowing a water evaporation at room temperature for a period of 4 days. Gelatin films (Ge) were performed in a similar way but without adding the crosslinking agent.

3.2.2 Crosslinking of gelatin films

To study the effect of crosslinking, a process will use dry gelatin films in a seal dessicator containing 25ml of aqueous glutaraldehyde solution in a Petri dish at 37°C. The degree of crosslinking can vary by controlling time periods.

3.3 Characterization and Testing:

3.3.1 Characterizations

3.3.1.1 Crosslinking density determination

In order to estimate the network crosslinking density, the number-average molecular weight of chain segments between two crosslinking points, *M*c, was calculated from equilibrium water uptake experiments performed at 20 ◦C, according to the Flory–Renher equation [8]:

$$Mc = \frac{\rho V1(\varphi^{1/3}_g - 2\varphi_g/f)}{X\varphi^2_g + \ln(1 - \varphi_g) + \varphi_g} \quad (1)$$

where ρ is the density of the dry gelatin determined by picnometry, $V1$ the molar volume of the solvent, χ the polymer–solvent interaction parameter and was taken from the literature [8] ($\chi = 0.49 \pm 0.05$) and φg is the volume fraction of the swollen gelatin. The gelatin volume fraction in the swollen samples (φ_g) was estimated assuming the following relationship:

$$\Phi_g = \frac{W_0 \rho w}{W\rho_g - W_0(\rho - \rho_w)} \quad (2)$$

where W_0 is the initial weight of the sample, W the weight of the swollen sample, ρ_w the density of the water at room temperature and ρ is the density of the dry and uncrosslinked gelatin film.

3.3.1.2 Thermogravimetric analysis (TGA)

A thermal gravimetric analyzer (DuPont, model TGA 2950) was used to determine the amount of moisture content and the decomposition temperatures with the temperature scan from 30 to 600 °C with a heating rate of 5 °C/min, for crosslinked film with % vol crosslinking agent of glutaraldehyde (0.5,1, 3, 5, 7) and non-crosslinked gelatin films. The samples were weighted in the range of 5–10 mg and loaded into a platinum pan, and then heated it under a nitrogen gas flow.

3.3.1.3 Melt Rheometer (Rheometric Scientific, ARES)

The electrorheological properties of the crosslinked and uncrosslink gelatins were investigated in terms of electric field strength. A melt rheometer (Rheometric Scientific, ARES) was fitted with a custom-built copper parallel plates fixture (diameter of 25 mm). A DC voltage was applied with a DC power supply (Instek,GFG8216A), which can deliver electric field strength to 1 kV/mm. A digital multimeter was used to monitor the voltage input. In these experiments, the oscillatory shear strain was applied and the dynamic moduli (G' and *G*") were measured as functions of frequency and electric field strength. Strain sweep tests were first carried out to determine the suitable strains to measure G' and *G*" in the linear viscoelastic regime. The appropriate strain was determined to be 0.2% for gelatin film samples. For the 3% crosslinked high gel strength gelatin film sample, the strain of 0.14% was used. The frequency sweep tests were carried out to measure G' and *G*" of each sample as functions of frequency. The deformation frequency was varied from 0.1 to 100 rad/s. Prior to each measurement, the non-crosslinked gelatin and the 3% high gel strength gelatin film samples were presheared at a low frequency (0.04 rad/s) under an electric field for 15 min to ensure the formation of equilibrium polarization before the G' and *G*" measurements. Experiments were carried out at the temperature of 27 °C and repeated at least two or three times. The effect of temperature was studied at various temperatures between 27 and 107 °C for the non-crosslinked gelatin film sample. The temporal response experiments were carried out at 1 kV/mm for the non-crosslinked gelatin and the 3% crosslinking high gel strength gelatin film samples.